NOTICE.

Future issues of this publication placed under either the "Special Veermary" or "Special Forest Series" will not be included in the annual enumeration. Such papers are printed for Departmental purposes. Their unfortunate inclusion in the system of annual numbering has led recipients of the ordinary issues to think their sets incomplete.

The following pamphlets have already appeared as Special issues, and have no accordingly been furnished to the public:—

1894 . Nos. 8, 9, 10, 11, 13 and 15.

1896 . No. 8.

(Medicinal Series, No. 12.)

THE

AGRICULTURAL LEDGER

1898-No. 3.

ACONITUM FEROX.

(INDIAN ACONITE.)

DICTIONARY OF ECONOMIC PRODUCTS, Vol. 1., A. 397-400.)

CONTRIBUTIONS TO OUR KNOWLEDGE OF THE ACONITE ALKALOIDS.

ON PSEUDACONITINE

By WYNDHAM R. DUNSTAN, M.A. F.R.S., and FRANCIS H. CARR, A.I.G., Salters' Company Research Fellow in the Laboratories of the Scientific Department of the Imperial Institute. Reprinted from the Transactions of the Chemical Society, 1897.

Other DICTIONARY articles that may be consulted a

Aconitum heterophyllum, Vol. I., A. 401. A. Napellus, Vol. I., A. 413.

Also

The Agricultural Ledger Nos. 32 of 1896; and 19 of 1897.



CALCUTTA:

OFFICE OF THE SUPERINTENDENT, GOVERNMENT PRINTING INDIA. 1898.

The objects of THE AGRICULTURAL LEDGER are-

- (1) To provide information connected with agriculture or with economic products in a form which will admit of its ready transfer to ledgers;
 - (2) To secure the maintenance of uniform ledgers (on the plan of the Dictionary) in all offices concerned in agricultural subjects throughout India, so that references to ledger entries made in any report or publication may be reading
 - estilised in all offices where ledgers are kept;

 (2) To admit of the circulation, in convenient form, of information on any subject connected with agriculture or economic products to officials or other persons interested therein;

(4) To secure a connection between all papers of interest published on subjects

relating to economic products, and the official Dictionary of Economic Products. With this object the information published in these ledgers will uniformly be given under the name and number of the Dictionary article which they more especially amplify. When the subject dealt with has not been taken up in the Dictionary, the position it very possibly would occupy in future issues of that work will be assigned to it.

(Medical and Chemical Series, No. 12.) (SKedicinal Scoducto.)

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Bu WYNDHAM R. DUNSTAN, M.A., F.R.S., and FRANCIS H. CARR, A.I.C., Salters' Company Research Fellow in the Laboratorics of the Scientific Department of the Imperial Institute. Reprinted from the Transactions of the Chemical Society, 1897.

In previous papers communicated to this Society, an account has Introductor been given of an investigation of the principal properties and decomposition products of the alkaloid aconitine, derived from the 100ts of Aconitum Napellus. The enquiry has since been extended to a similar examination of the alkaloids occurring in other species and varieties of aconite. At the request of the Government of India, an investigation is being made, in the Scientific Department of the Imperial Institute, of the alkaloidal constituents of the chief kinds of aconite indigenous to India, especially of those which are highly posonous, or are reputed to be of medicinal value. In this connection, Dr. H. A. D. Jowett has described (Part XIII of this series) the principal properties and decomposition products of atisine derived from the Aconitum heterophyllum of India. In a previous communication, and in the present paper, we give an account of pseudacomitine, the highly poisonous constituent of the acomite occurring in Nepaul, which is usually regarded as Aconitum ferox, and locally known as "bish" (bikh). Our previous knowledge of this alkaloid salmost wholly due to the researches of Alder Wright, who, in conjunction with Luff, gave an account of its properties in a paper communicated to this Society in 1878. The material employed in our

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The Accelte

PREUDACONI

work consisted of roots of the plant, which were specially collected with great care in the Himálayas under the supervision of Dr. George Watt, C.I.E., the Reporter on Economic Products to the Government of India.

Pseudaconitina.

Already

of this

Best solvent for extraction

tion

Examination of crystals. Result. In a preliminary notice communicated to the Society two years ago (Proc., June, 1895) the authors described some of the properties of pseudaconitine. They showed that its hydrolysis occurs in the stages, in the first of which acetic acid and a crystalline base versury, pseudaconine are formed, and in the second the elimination of a molecule of dimethylprotocatechnic acid takes place with the formation of pseudaconine. It has also been shown that, when pseudaconitine is heated in the dry state, one molecular proportion of acetic acid distils over, and a base is left, to which the name pyropseudaconitine was given. This base, when hydrolysed, for nishes dimethylprotocatechnic (veratric) acid and pyropseudaconized. The present paper gives a more detailed account of the experiment which furnished these results, and also an account of other observations on the properties of the salts and derivatives of pseudaconitine.

Extraction of the Alkaloid.—Several methods have been tree for the extraction of the base from the root, involving the use of methylic, ethylic, and amylic alcohols. Finally, a mixture of methylic and amylic alcohols, in the proportion of 5 to 1, was adopted as the most efficient solvent. The methylic alcohol is distilled from the slightly warmed percolate, under reduced pressure, when a quantity of fat separates; this is removed, and the alkaloid is extracted from the amylic alcohol by shaking it with very dilute (1 per cent.) aqueous hydrochloric acid. The solution is then shakea with ether, to remove the dissolved amylic alcohol, the alkiled liberated by the addition of dilute ammonia, and then extracted by shaking with ether in the usual manner. On evaporating the dried ethereal solution, white crystals separate, which are recristallised by dissolving them in dry chloroform, adding dry ether. and then dry light petroleum, until a slight turbidity is produced: by this means, a considerable supply of pure pseudaconitine was obtained. Judging from the yield obtained from the roots of Aconitum ferox, it would appear that more pseudaconitine is present in them than there is of aconitine in the roots of A. Napellus; but this is a question to which we shall return in a future paper.

Properties of Pseudaconitine.—The pure base crystallises well Mr. W. J. Pope has kindly examined some fairly well-defined crystals.

with the following results.

"The crystals of pseudaconitine consist of small, colourless transparent crystals of rhomboidal shape having a rather vitreous lustre. Owing to the poor character of the images obtained from the various faces, the measurements given below are of no great accuracy; they would indicate that the crystals belong to the orthorhombic system. That the crystals are, however, not orthorhombic, is shown by the faces which they exhibit, and also by the interference

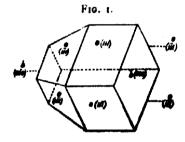
ACONITU ferox.

figure observed in polarised light. Considering, for the purpose of PSEUDACO description, that the crystals are really orthorhombic, the following faces are always observed: -(010), (070), (111), (117), (171), (171), (TTT), and (TT1), with traces of the form {110}; the two faces (T11) and (T1T) are never observed. This observation was made on all the crystals examined—ten in number, belonging to two different crops—and the same faces of the forms [010] and [111] were found in every case; this arrangement of faces is not possible in the hemihedral subdivisions of either the orthorhombic or monosymmetric system. The crystals must, therefore, be assigned to the rare anorthic hemihedral system, two of the forms having the indices 111 being present as half-forms only, and the interaxial angles a, B, and y being equal to 90° within the rather wide limits of error incurred in the measurement of crystals such as those now described.

"The crystals present the appearance shown in Fig. 1, and have

the axial ratios-

$$a:b:c=0.8362:1:0.6938.$$



"The following angular measurements were obtained:—

	Angle,	No. of measurements.	Limits.	Mean.	Calculated.
ès	= 010 : [11	23	61° 14' - 62° 17	61° 54'	
54	# III : III	10	54 27 - 56 46	56 5	56" 12"
P	= olo : 111	9	117 26 118 19		118 6
	= 111 : 111	18	67 12 - 68 59		-
84	= 111 : 111	7	111 14 - 111 35	111 24	111 26
	= 111:111	8	94 1 - 95 29	94 35	91 29
	= 111:111	4	85 2 - 85 37	94 35 85 20	85 31
	= 110: 110	4	78 54 - 80 16		79 48
Ħ	= 110:110	i	_	101 4	100 12

"The crystals are very brittle, and possess a good cleavage; the latter, however, could not be determined. On examining a cleavage fragment under a very wide angle objective, one optic axis is seen to emerge at the edge of the field; it shows that the dispersion is inclined, which is only possible in the monosymmetric or anorthic

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system. The hemihedral character of the crystals is of interest because non-superposable hemihedrism is so rarely observed on crystals of the natural alkaloids, that it has been said not to occur.

(Wysouboff, Ann. Chim. Phys., 1894. [vii], 1, 11.)
"The crystalline form of aconitine has been determined by Tutton (Trans., 1891, 59, 288), who found the crystals to be orthorhombic, but did not observe hemihedrism. Although morphotropic relationships would seem to exist between the crystalline forms of aconitine and pseudaconitine, yet these can hardly be worked our from the data now given for the latter alkaloid; the following

corresponding angles on the two compounds seem to show some

similarity.

4.01	
	16
1	

The crystals melt with decomposition at 201°, acetic acid grade-

Pronduconities.

Melting point.

Salvents.

ally distilling off; the melting point is fairly sharp if the substance is put into the bath heated to 150° and the temperatures slowly raised Wright and Luff (loc. cit.) have recorded 104—105° as the melting point of pseudaconitine. They state that the alkaloid contains 1H₄O. which is lost at 100°, but we have not been able to confirm this observation. Pseudaconitine dissolves readily in alcohol, chloroform. and acctone, less readily in ether, very slightly in water, and scarcely

at all in light petroleum. Pseudaconitine is dextrorotatory. A determination of the specific rotatory power using an alcoholic solution, gave-

$$c = 1.12$$
 $l = 2 dm$, $a = 25$ $t = 15$
whence $[a]_0 = \frac{100 \times 25}{2.1 \times 1.12 \times 60} = +18^{\circ} 36^{\circ}$.

The ordinary salts of pseudaconitine are lavorotatory, and usually soluble in water and alcohol. Combustion of the alkaloid made with the material dried at 100° gave the following results-

l. 0'2012 gave 0'5964 CO, and 0'1691 H₁O. C = 62'29; H = 7'20. II. 0'2587 " 0'5975 CO, " 0'1484 H₂O. C = 62'96; H = 6'37.

These figures nearly correspond with those calculated from the formula proposed by Wright and Luff (Trans., 1878, ii, 151), namely, Commiss proposed by wright and Luff (Trans., 1878, ii. 151), namely, Coth 180, NO. 181, for which the calculated percentages are, for carbon, 63-88; for hydrogen, 713. Like aconitine, pseudaconitine and its salts, even in very dilute solution, give rise to a persistent tingling and numbing sensation on the tongue, and are highly poisonous. From preliminary experiments on the relative toxicity of various aconitie alkaloids, which have been made at our suggestion by Dr. F. W. Tunnloilffs, it would appear that pseudaconitine is slightly more toxic than aconitine. more toxic than aconitine.

ACOMITUN ferox.

Salts of Pseudaconitine.

Preudaconitine hydrochloride, C., H., NO,, HCl.—We have not not obtained the sale in a crystalline condition. It has succeeded in obtaining this salt in a crystalline condition. It has been prepared by the direct action of dilute hydrochloric acid on both aqueous and alcoholic solutions of the base, but all attempts to crystallise it from water, alcohol, or a mixture of alcohol and ether have resulted in the production of a colourless varnish.

Prendecontline hydrobromide, C3.4H.2NO1.4.HBr.—This salt is pre-pared by dissolving the base in dilute hydrobromic acid and evaporating the solution. A colourless varnish remains, and on adding a limle alcohol to this, the mass rapidly becomes crystalline. It is best purified by dissolving it in dry alcohol and adding dry ether until a slight turbidity is produced: it then separates in large, cubical crystals often arranged in rosettes. The salt readily dissolves in alcohol and water, but is insoluble in ether and light petroleum. The crystals contain 2H, O, which are expelled on drying at 100-103°. The dried substance melts at 191". The water of crystallisation was estimated by heating at 100-103° in an air bath-

0'546 lust 0'0263 = 4'8 per cent.
$$H_1O$$
.

Determinations of the bromine in the undried and in the dried substance gave the following figures-

o 3370 undried salt gave 0'0751 AgBr. Br = 9'44. 05107 dried salt gave 01227 AgBr. Br = 1005. Call NOm HBr + 2HO requires H2O = 4'5. Br = 9'95 per cent. CultunOm HBr requires Br = 103 per cent.

An agreeous solution of the salt is lavorotatory; the determination of the specific rotatory power led to the following result-

Pseudaconitine, therefore, resembles aconitine in being a dextrorotatory base whose salts are lævorotatory.

Pseudaconitine hydriodide, C. H., NO., HI. - This salt is precipitated in an amorphous condition when aqueous potassium todide is added to an aqueous solution of pseudaconitine hydrobromide. Although at first amorphous, the precipitate rapidly becomes crystalline; it may readily be purified by recrystaltisation from a mixture of alcohol and ether.

Pseudaconstine nitrate, C. . H. . NO , . HNO , . This sait was prepared by Wright and Luff (iec. cit.) by dissolving the base in dilute nitric acid, and precipitating the nitrate by adding strong nitric acid, in which it is only sparingly soluble; this method, however, is not to be recommended, as strong nitric acid is very apt to decompose the alkaloid. By dissolving the alkaloid in dilute nitric acid to exact neutrality, and evaporating to dryness, the nitrate is obtained as an amorphous varnish, which crystallises at once on the addition

Bromins.

Similarity to

The Mitrata.

CONITUM ferox.

of alcohol; it is readily purified by crystallisation from a mixture of alcohol and ether, and when pure may be crystallised from water. The dried salt melts at 1920 and effervesces at a slightly higher temperature; the melting point is fairly sharp if the substance is put into the bath at 155° and slowly beated. The water of crystalli-sation was determined by heating the powdered, air-dried salt at 100-105°.

0'1975 lost 0'014 H2O. H2O = 7'0 per cent. CaHaNOs HNOs + 3H2O requires H2O = 67 per cent.

Its solubility in water at 150 was determined. 5 c.c. of a solution saturated at this temperature yielded, on evaporation, 0.300 gram of salt. 100 c.c. of water at 15°, therefore, dissolves 4'18 grams of salt.

Hydrolysis of Pseudaconitine.

Acutic seld.

We have previously pointed out (loc. cit.) that, in addition to the pseudaconine and veratric acid, observed by Wright and Luff, acetic acid is formed by the hydrolysis of pseudaconitine, and we have also shown that the hydrolysis may occur in two stages. To determine the first stage only in the hydrolysis, namely, the elimination of acetic acid with the formation of veratrylpseudaconine, it is best to employ a process similar to that which was found to answer in the case of aconitine. A neutral aqueous solution of a pseudaconitine salt, preferably the sulphate, is heated in a sealed tube at 135° for 3 hours, the amount of acetic acid formed is determined by direct titration with N/10 alkali, and the alkaloid, after being liberated by the addition of sodium carbonate, is dissolved by shaking with ether,

A solution containing o'168 gram of alkaloid (as salt), after this treatment, required for neutralisation 4'3 c.c. of N/10 alkali = 7'5 per cent, of acetic acid, which is slightly lower than that calculated for

one molecular proportion, namely, 8.7 per cent.

Analysis of the silver salt of this acid showed that it contained 64'56 per cent. of silver. Silver acetate contains 64'66 per cent. The formation of veratrylpseudaconine may thus be represented by the following equations—

 $C_{in}H_{ip}NO_{js} + H_{s}O = C_{js}H_{ip}NO_{js} + CH_{s}COOH.$ Pseudaconitine Veratrylpseudaconine.

Veratrylpseudaconine.

Melting noint.

Sliver sait.

The pure base crystallises from ether in large, irregular crystals. which are nearly insoluble in water and in light petroleum, but readily soluble in ether, alcohol, and chloroform. They melt at 190 when put into the bath at 150. A solution of the base is lavorotatory. A determination of the specific rotatory power in alcoholic solution led to the following results-

$$t = 16^{\circ}$$
, $a = -1^{\circ}16' c = 1.5035$ $l = 2.2 dm$,
whence $[a]^{0} = \frac{100 \times 1.267}{2.2 \times 1.5035} = -38^{\circ}18'$

Veratrylpseudaconine, therefore, unlike its analogue benzaconine, exhibits rotatory power of the opposite sign to that of its parent base. Aconitine and benzaconine are both dextrorotatory, whilst pseud-

aconitine is dextrorotatory and verstrylpseudaconine layerotatory. Combustions of the base, dried at 100-103°, furnished the follow-

I. Carbon 61:44; hydrogen 7:15 per cent. broi: 7'05 ...

Calculated for Ca, H., NO, H.O: Carbon 61:54; hydrogen 7:39

This alkaloid and its salts have a very bitter taste, but produce no

tingling sensation, and do not appear to be poisonous.

ing results, showing it to be a monhydrate-

Veratry I pseudaconine Hydrobromide, C. HaNO ... HBr. - This salt separates from a mixture of alcohol and ether in large, prismatic crestals which contain 311.0-

Absence of properties

Meiting points

or3478 salt lost or0264 H_2O at 100°. $H_3O = 7.5$. 0.34/8 ... gave 0.0842 AgBr. Br = 11/21. C_HH_GNO₁₀HBr + 3H₂O requires H₂O = 6/954 Br ==11/02 per cent.

The salt is soluble in water, alcohol, and chloroform,

Veratrylpseudaconine nitrate, Cx. H., NOx. HNOx. crystallises from a mixture of alcohol and ether in rhombic prisms arranged in rosettes. In melting, two fairly sharp points may be noticed, one at 222°, when softening and change commences, and at 232°, when the salt melts sharply with decomposition. Combustion of the anhydrous

salt gave C=57'34; H=6'40 per cent.: C₃₄H₄₇NO₃₄HNO₃ requires C=57'62; H=6'77 per cent. Veratry Ipseudaconine aurichloride, C.H., NO., HAuCl. thrown down as a pale yellow, amorphous precipitate when auric chloride is added to a solution of the hydrochloride. It is insoluble

in water, ether, and light petroleum, but readily soluble in ethylic and methylic alcohols, chloroform, and acetone; it could not be crystallised from any of the last-mentioned solvents alone, or on the addi-

tion of any of the former to them.

Pseudaconine.

The second stage of the hydrolysis by which veratrylpseudaconine passes into veratric acid and pseudaconine, may be best effected by adding alcoholic soda to an alcoholic solution of pseudaconitine, or veratrylpseudaconine. Hydrolysis takes place rapidly, and is complete in about a hours. Dilute sulphuric acid is then added, the filtrate evaporated, the veratric acid extracted from the acidified solution by ether, and the pseudaconine by chloroform, after rendering the solution alkaline with ammonia.

0'2143 gram of alkaloid gave 0'0556 gram of veratric acid=25'94 per cent. Calculated for one molecular proportion, 20:49 per cent.

Hydrolysis

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The acid melts at 178° and exhibits the other properties of dimethylprotocatechuic acid (veratric acid). This stage of the hydrolysis may, therefore, be represented by the equation—

C₃₁H₁₇NO₃₁ + H₂O = C₈H₂(OCH₂)₇COOH + C₃₁H₂₀NO₈ Veratrylpseudaconine Veratric acid Paeudaconine.

Preudaconine is an amorphous, hygroscopic base readily soluble in water, chloroform, alcohol and acetone, and less readily in ether. Its aqueous solution is strongly alkaline to litmus. All attempts to crystallise the base uncombined with its solvent have been unsuccessful.

An aqueous solution of pseudaconine is dextrorotatory. The specific rotatory power of an aqueous solution was determined with the following results—

$$a[20^{\circ}] = +32'5$$
 $c = 0.800$ $l = 2 dn$
whence $[a] = \frac{100 \times 0.541}{2 \times 0.890} = +30'' 6'$.

Attempts at crystallisation Pseudaconine hydrachloride, C₃₃ H₁₀ NO₄₀ HCl, was prepared by dissolving the base in dilute hydrochloric acid to neutrality. Many attempts made to crystallise this salt from various solvents were unsuccessful, although, on one occasion, crystals were obtained from an alcoholic solution which had stood for six months; these were prisms and melted at 68°.

Pseudaconine hydrobromide, C_{2.3}H₃₀NO₈,HBr, was prepared in the same manner as the hydrochloride, but it could not be crystallised

Pseudaconine mitrate, C₁₆H_{sp}NO_n, HNO₃, was prepared by the direct action of dilute nitric acid on the base, and also by double decomposition between silver nitrate and the hydrochloride, and barium nitrate and the sulphate. It was always obtained in an amorphus state.

Pseudaconine sulphate, (C_{3.5}H_{3.0}NO₃)₃, H₃SO₄, was prepared by acting on pseudaconine with dilute sulphuric acid, but this salt could not be crystallised.

Pseudaconine aurichloride, C_{nn}H_{nn}NO_n.HAuCl_n, is precipitated when auric chloride is added to a concentrated solution of pseudaconine hydrochloride. It is a yellow, amorphus precipitate sparingly soluble in water, and could not be crystallised from any of its solutions. When light petroleum is used, the yellow colour of the solution is discharged and it becomes colourless, although no gold is precipitated. This change, in other cases, has been observed to be due to the production of an aurichlor-derivative by loss of hydrogen chloride from the aurichloride. We have so far failed to crystallise an aurichlorpseudaconine from this solution.

Pyropseudaconitine.

Behaviour when heated above meltAs previously recorded by us, when pseudaconitine is heated slightly above its melting point, it effervesces and loses acetic acid. A determination of the amount of acetic acid which distils under these

circumstances, proved that one molecular proportion of acetic acid is expelled; analysis of the silver salt of the acid proved it to be silver acetate. The reaction may, therefore, he represented by the following equation-

> $C_mH_mNO_{13} = C_1H_1O_1 +$ CaHaNOa Pyropseudaconitine. Pseudaconitine

Pyropseudaconitine, an anhydride of veratrylpseudaconine, is obtained from the residue by solution in dilute acid, and is purified by fractional precipitation from this solution with dilute ammonia. The colourless fractions are dissolved in dilute acid, precipitated with ammonia, and the pure base extracted from the alkaline solution by ether. The base so far has only been obtained as an amorphous varnish, nearly insoluble in water, but readily soluble in alcohol, chloroform, and ether. Its salts appear to crystallise well; they have a bitter taste, but produce no tingling, and do not seem to be poisonous. The hydriodide crystallises in prisms.

Although, in publishing our preliminary notice of pseudaconitine we stated that we were engaged in a complete investigation of the alkaloid, this did not prevent Herr Martin Freund from examining the alkaloid, and, nine months after the appearance of our paper in the Proceedings of this Society, publishing in the "Berichte" (Ber., 20. 6. 853) an account of his and Herr Niederhoffheim's experiments on the subject. They adopt Wright's formula for the alkaloid, and confirm our conclusion that pseudaconitine, like aconitine, contains an acetyl group. For the rest, they record melting points which differ somewhat from those previously recorded by us, but since these points are in most cases decomposing points, and depend on the conditions under which the observations are made, no real importance attaches to these discrepancies.

As to their assertion that pseudaconine is the anhydride of the aconine derived from aconitine, it is to be observed that this statement is based solely on the numerical coincidence that the formula for pseudaconine deduced from Wright's formula for pseudaconitine. namely, C_{1.5}H_{2.9}NO_{1.8} differs by one molecule of water from the formula which Fround has suggested for aconine (C_{2.5}H_{4.8}NO₂). But, as we have elsewhere pointed out, Fround's new formulæ for aconitine and its derivatives cannot at present be accepted as proved, and we have so far seen no reason to depart from a formula for aconiune, differing very slightly from that originally suggested by Wright, which does not allow of pseudaconine $(C_{\pi,1}H_{\pi,n}NO_{\pi})$ being regarded as the anhydride of aconine $(C_{+4}H_{3,9}NO_{10})$. As a matter of fact, we have already described an anhydride of aconine (pyracotime), whose properties are very different from those of pseudaconine.

We have pleasure in acknowledging the skilful help afforded us in the early stages of this work, in the Research Laboratory of the Pharmacentical Society, by Mr. H. T. Durant.

All communications regarding THE AGRICULTURAL LEDGER should be addressed to the Editor, Dr. George Watt, Reporter on Economic Products to the Government of India, Calcutta.

the objects of this publication (as already stated) are to gradually develop and perfect our knowledge of Indian Agricultural and Economic questions. Contributions or corrections and additions will therefore be most welcome.

In order to preserve a necessary relation to the various Departments of Government, contributions will be classified and numbered under certain series. Thus, for example, papers on Veterinary subjects will be registered under the Veterinary Series; those on Forestry in the Forest Series. Papers of more direct Agricultural or Industrial interest will be grouped according as the products dealt with belong to the Vegetable or Animal Kingdom. In a like manner, contributions on Mineral and Metallic subjects will be registered under the Mineral Series.

This sheet and the title-page may be removed when the subject-matter is filed in its proper place, according to the letter and number shown at the bottom of each page.

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